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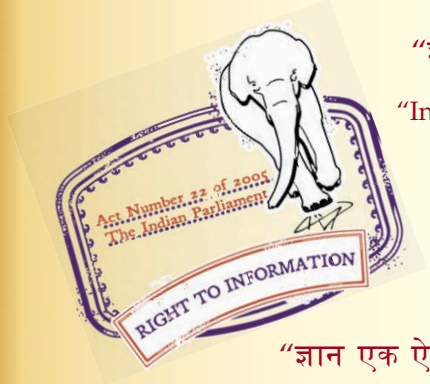

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METHODS FOR MEASUREMENT OF EMISSIONS FROM STATIONARY SOURCES

PART 2 SULPHUR DIOXIDE

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PART 2 SULPHUR DIOXIDE

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METHODS FOR MEASUREMENT OF EMISSIONS FROM STATIONARY SOURCES

PART 2 SULPHUR DIOXIDE

0. FOREWORD

0.1 This Indian Standard was adopted by the Indian Standards Institution on 28 March 1985, after the draft finalized by the Air Quality Sectional Committee had been approved by the Chemical Division Council.

0.2 Practically all fuels in common use contain variable amounts of sulphur, most of which is discharged to the atmosphere as sulphur dioxide during combustion. In addition, specific industrial processes produce large quantities of sulphur dioxide, some of which may escape into the air. Because it is known to be potentially harmful both from health and economic point of view, it is necessary to regulate the emissions of sulphur dioxide.

0.3 In reporting the result of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS : 2-1960*.

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1. SCOPE

1.1 This standard prescribes the IPA-Thorin method for measurement of sulphur dioxide emissions from stationary sources.

2. TERMINOLOGY

2.1 For the purpose of this standard, definitions given in IS : 4167-1980† shall apply.

3. PRINCIPLE

3.1 A gas sample is extracted from the sampling point in the stack. The acid mist, including sulphur trioxide, is separated from the sulphur dioxide and the sulphur dioxide fraction is measured by the barium thorin titration method.

*Rules for rounding off numerical values (*revised*).

†Glossary of terms relating to air pollution (*first revision*).

4. RANGE AND SENSITIVITY

4.1 The procedure given is designed to cover high concentration of sulphur dioxide. Though the minimum value recommended is 1 ppm, this method gives better result at higher concentration.

5. INTERFERENCES

5.1 There is no interference due to nitrates, chlorides, fluorides, bicarbonates, hydrogen peroxide, etc. The major interference expected is from cationic species like K^+ and Na^+, which are not present in most of the sources sampled and may be removed by using a heated, high efficiency, glass fibre filter before the impinger. This technique can also be used when sulphur dioxide concentration is low.

6. APPARATUS

6.1 Apparatus for Sampling

6.1.1 *Probe* — Chemical resistant glass, 5 to 6 mm ID, with a heating system to prevent condensation and filtering medium to remove particulate matter including sulphuric acid mist.

6.1.2 *Dust Trap* — For low dust concentration (up to 1 g/m^3N) are a standard large impinger with glass wool packed in top to prevent acid mist carry over. For high dust concentrations, use an appropriate thimble.

6.1.3 *Impingers* — Three standard large impingers [*see* IS : 5182 (Part 5) - 1975*].

6.1.4 *Drying Tube* — Packed with 1-3 mm size indicating type silica gel, or equivalent, to dry the sample.

6.1.5 *Valve* — Needle valve or equivalent, to adjust flow rate accurately in the range of 2-5 l/min.

6.1.6 *Pump* — Leak-free, vacuum type.

6.1.7 *Rotameter* — Rotameter or other suitable device, to measure flow rate in the range of 0-10 l/min.

6.1.8 *Dry Gas Meter* — Sufficiently accurate to measure the sample volume within 1 percent.

6.2 Apparatus for Sample Recovery

6.2.1 *Glass Wash Bottles* — Two.

6.2.2 *Polyethylene Storage Bottles* — To store impinger samples.

*Methods for measurement of air pollution: Part 5 Sampling of gaseous pollutants.

6.3 Apparatus for Analysis

6.3.1 *Pipettes* — Transfer type, 5-ml and 10-ml sizes (0·1 ml divisions) and 25-ml size (0·2 ml divisions).

6.3.2 *Volumetric Flasks* — 50-ml, 100-ml and 1 000 ml.

6.3.3 *Burettes* — 5 ml and 50 ml.

6.3.4 *Long-necked Flask* — 125 ml.

7. REAGENTS

7.1 Reagents for Sampling

7.1.1 *Water* — Deionized or distilled water is preferable for sharp end points.

7.1.2 *Iso-propanol, 80 percent* — Mix 80 ml of *iso*-propanol with 20 ml of distilled water.

7.1.3 *Hydrogen Peroxide, 3 percent* — Dilute 100 ml of 30 percent hydrogen peroxide to 1 litre with distilled water. Prepare fresh daily.

7.2 Reagents for Sample Recovery

7.2.1 *Water-Deionized or Distilled* — Deionized water is preferable for sharp end points.

7.2.2 *Iso-propanol, 80 Percent*

7.3 Reagents for Analysis

7.3.1 *Water-Deionized or Distilled* — Deionized water is preferable for sharp end points.

7.3.2 *Iso-propanol*

7.3.3 *Thorin Indicator* — 1 (O-arsonophenylazo)-2-naphthol-3, 6-disulfonic acid, disodium salt (or equivalent). Dissolve 0·20 g in 100 ml distilled water.

7.3.4 *Barium Perchlorate (0·01 N)* — Dissolve 1·95 g of barium perchlorate $Ba(ClO_4)_2 . 3H_2O$ in 200 ml distilled water and dilute to 1 litre with *iso*-propanol. Standardize with sulphuric acid. Barium chloride may be used.

7.3.5 *Sulphuric Acid, Standard (0·01 N)* — Standardize to $\pm 0{\cdot}0002$ N against 0·01 N NaOH which has previously been standardized against potassium acid phthalate (primary standard grade).

8. PROCEDURE

8.1 Preparation of Collection Train — Pour 15 ml of 80 percent *iso*-propanol into the impinger and 15 ml of 3 percent hydrogen peroxide into each of the first two impingers. Leave the final impinger dry. Assemble the train as shown in Fig. 1. Check the sampling train for leakage at the sampling site by plugging the probe inlet and pulling a vacuum corresponding to 250 mm mercury column. A leakage rate not in excess of 1 percent of the sampling rate is acceptable. Carefully release the probe inlet plug and impingers and add more ice during the run to keep the temperature of the gases leaving the last impinger at 20°C or less.

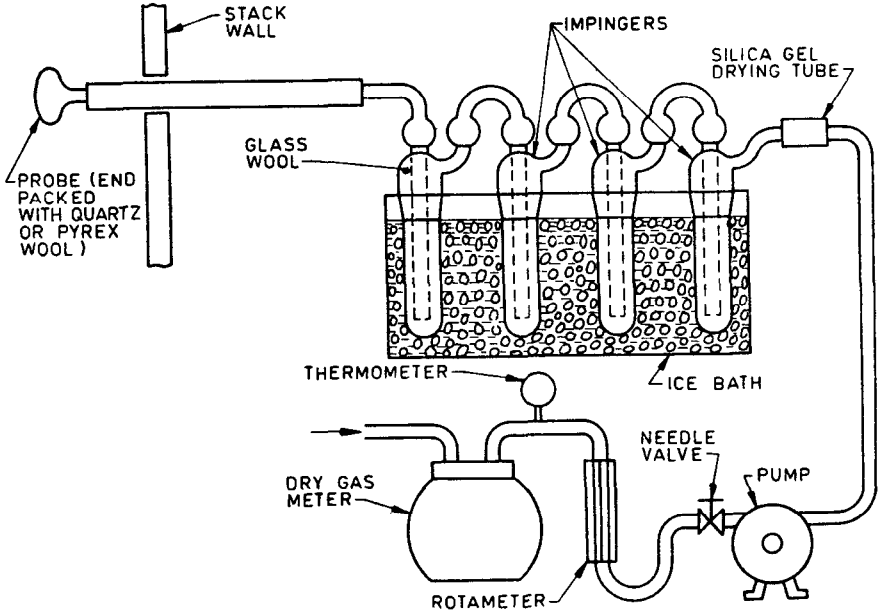

FIG. 1 SULPHUR DIOXIDE SAMPLING TRAIN

8.2 Sample Collection — Adjust the sample flow rate in the range 2 to 5 litres/minutes. To begin sampling, position the tip of the probe at the first sampling point and start the pump. At the conclusion of each run, turn off the pump and record the final readings. Remove the probe from the stack and disconnect it from the train.

8.3 Sample Recovery — Disconnect the impingers after purging. Discard the contents of the mist impinger (with the glass wool). Pour the contents of the other impingers into a polyethylene shipment bottle. Rinse the

three midget impingers and the connecting tube, with distilled water and add these washings to the same storage container.

8.4 Sample Analysis — Transfer the contents of the storage container to a 50 ml volumetric flask. Dilute to the mark with deionized, distilled water. Pipette a 10 ml aliquot of this solution into a 125 ml erlenmeyer flask. Add 40 ml of *iso*-propanol and two to four drops of thorin indicator. Titrate to a pink end point using 0·01 N barium perchlorate. Run a blank with each series of samples.

9. CALIBRATION

9.1 Use methods and equipment which have been approved to calibrate dry gas meter, and rotameter.

9.2 Standardize the barium perchlorate against 25 ml of standard sulphuric acid containing 100 ml of *iso*-propanol.

10. CALCULATIONS

10.1 Dry Gas Volume — Correct the sample measured by the dry gas meter to normal conditions (298 K and 101 kPa) by using the following equation:

$$V_N = \frac{V(T_N) \times (P)}{(T) \times (P_N)}$$

where

V_N = volume of gas sample through the dry gas meter (normal conditions), m^3;

V = volume of gas sample through the dry gas meter (meter conditions), m^3;

T_N = absolute temperature at normal conditions, (289 K);

T = average dry gas meter temperature, K;

P = absolute meter pressure, kPa; and

P_N = absolute pressure at normal conditions, kPa (101 kPa).

10.2 Sulphur Dioxide Concentration — Calculate the concentration of sulphur dioxide using the following equation:

$$C = 0{\cdot}032 \times \frac{(V - V_b) \times N \times \frac{V_{s0}}{V_a}}{V_N}$$

where

V = volume of barium perchlorate titrant used for the sample, ml;

V_b = volume of barium perchlorate titrant used for blank, ml;

N = normality of barium perchlorate titrate, g-eq/l;

V_{s0} = total solution volume of sulphur dioxide, ml;

V_a = volume of sample aliquot titrated, ml; and

V_N = volume of gas sampled through the dry gas meter (normal conditions), m^3.

10.3 Sulphur Dioxide Emissions — Calculate the emission of sulphur dioxide as follows:

$$E = C \times O_n \text{ g/h}$$

where

C = concentration of sulphurdioxide, g/m^3 (normal),

O_n = flue gas flow rate, wet conditions, m^3/h (298 K, 101 kPa),

NOTE — O_n is determined in accordance with IS:11255 (Part 3)-1985*.

11. PRECISION AND ACCURACY

11.1 The accuracy (sampling and analysis) of this method is ± 2 percent at 2000 ppm and ± 5 percent at 200 ppm. Interferences are minimal and yields precision better than ± 3 percent.

*Methods for measurement of omissions from stationary sources: Part 3 Flow rate.

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